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**(54) Stabilized pumpable aqueous dispersions**

(57) A method of rendering readily pumpable but stable an aqueous suspension of mineral particles such as whiting, kaolin or limestone, having a water content of not more than 40

per cent by weight, by incorporating an agent which acts in the presence of the solid particles to form a gel-like or thixotropic structure which inhibits settlement of the solids on standing but breaks down under gentle shear, and preferably also a dispersing agent capable of reducing the viscosity.

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## SPECIFICATION

### Aqueous suspensions

This invention relates to aqueous suspensions of particulate minerals for use in the preparation of water-borne adhesives, paints, and surface coating compositions for paper and the like.

More particularly the invention is concerned with aqueous suspensions of high solids content which have a reduced tendency to settlement during transport and storage before use.

Particulate minerals such as kaolins, and whittings prepared from limestone or natural chalk are widely used as pigments or extenders in water-borne coating compositions for application to a variety of surfaces such as paper, and exterior and interior wall surfaces.

For use in such coating compositions the mineral pigment or extender usually needs to be of very fine particle size. For instance whiting for use in water-borne paper coating compositions should contain not more than 0.5 per cent by weight of particles retained on a 45  $\mu\text{m}$  aperture sieve and at least 30 per cent by weight, more generally at least 40 per cent of particles smaller than 2  $\mu\text{m}$  equivalent spherical diameter. Materials of this degree of fineness can most conveniently be produced by wet methods of classification and/or comminution.

It would be of economic advantage if aqueous slurries of particulate mineral pigments produced in this way could be transported and incorporated direct into water-borne coating compositions, thus saving the cost and effort of drying the pigment and subsequently reslurrying or redispersing it in the water based coating composition.

For the transport and use of such particulate pigment in slurry form to be economically attractive it is necessary to keep the water content of the slurry as low as possible. The slurry should also be stable and not settle out during transport and storage before use and it should be pumpable.

It is known to reduce the viscosity of aqueous slurries of particulate minerals such as kaolin and whiting by the incorporation of small amounts of dispersing or surface active agents. By such means it is possible to achieve pumpable slurries containing as little as 30 per cent by weight of water or less depending on the nature and fineness of the particulate mineral particles and the amount and type of agent used. The amount of dispersing agent required to achieve a pumpable slurry of maximum solids content will usually be in the range 0.05 per cent to 0.50 per cent by weight of particulate solids. Agents which are conventionally used for this purpose include water soluble polyelectrolytes such as salts of polyacrylic and polymethacrylic acids, water soluble polyphosphates and polysilicates, lignosulphonates and alkyl aryl sulphonates.

A disadvantage of these water soluble agents is that the high solids slurries prepared with the aid of dispersing agents tend to settle out during transport and storage and the settled solids form a stubborn sediment which is less readily

redispersed than sediment arising in a slurry without such agents.

It is an object of the present invention to provide suspensions of mineral pigment particles of low water content that will be stable during transport and storage before use in the production of paper coating and other water-borne coating compositions.

According to the present invention there is provided a method of rendering readily pumpable but stable an aqueous suspension of mineral particles having a water content of not more than 40 per cent by weight, preferably not more than 35 per cent by weight, of the suspension, by incorporating in the suspension a structuring agent as herein defined as well as a small proportion of conventionally used dispersing agent of the aforementioned types. Preferably the size distribution of the mineral particles is such that not more than 0.5 per cent by weight of particles is retained on a 45  $\mu\text{m}$  mesh sieve and at least 25 per cent more preferably at least 60 per cent by weight of the particles are of size less than 2  $\mu\text{m}$  equivalent spherical diameter.

The mineral particles may for instance be of chalk whiting, kaolin or limestone.

The invention also embraces an easily pumpable aqueous mineral pigment suspension produced by the abovementioned method, and paper coating compositions in which all or part of the mineral pigment content is provided in the form of such a suspension.

In the paper coating industry it is generally acceptable that a high solids content slurry can be considered pumpable if its viscosity at shear rates of 100—500  $\text{sec}^{-1}$  is less than 2 poise, although it is possible to pump, under suitable conditions, slurries up to 20 poise.

By structuring agent is meant a material which when added to a suspension of particulate solid matter in water acts in the presence of the solid particles to form a gel-like or thixotropic structure which inhibits settlement of the particulate solids on standing but which breaks down under the influence of gentle shear so allowing the suspension to become readily pumpable.

Structuring agents which have been shown to provide the required stability when used in combination with a dispersing agent are synthetic hectorites e.g. the 'Laponite' (registered Trade Mark) products manufactured by Laporte Industries Ltd., titanium esters such as triethanolamine titanate, silica sols e.g. Ludox DM30 (registered Trade Mark) manufactured by E.I. du Pont de Nemours & Co., Inc., polyvinyl alcohols (of MW 14000 to 125000), carboxymethylcellulose, casein, and quaternary ammonium salts.

The amount of structuring agent required to be added to provide the necessary degree of stability to a suspension of particulate mineral pigment will depend on the nature and fineness of the mineral pigment particles and the water content of the suspension but will usually be in the range of 0.01 per cent to 1.5 per cent by weight of the solid

particles. A surprising feature is that when a structuring agent is added in this way the amount of dispersing agent needed to reduce the suspension viscosity can be considerably reduced as compared with the amount used above. In some cases the structuring agent may completely eliminate the need for a dispersing agent but whether or not this is done will depend also on economic considerations.

A preferred combination of agents is tetrasodium pyrophosphate as dispersing agent and synthetic hectorite as structuring agent.

The rheological properties of a suspension produced in accordance with the method of the invention and containing not more than 0.5 per cent by weight of particles retained on a 45  $\mu\text{m}$  aperture sieve, not more than 40 per cent by weight of water, from 0.01 per cent to 1.0 per cent by weight of solid particles of a conventionally used dispersing agent and 0.01 per cent to 1.5 per cent of a structuring agent of one of the aforementioned types are typified by a thixotropic nature such that in the absence of shear the pigment particles are held in suspension as a gel-like structure. Under slight shear this structure is destroyed so that the suspension becomes mobile and can be easily pumped. Once the shear is removed, as when the suspension is to be stored, the gel structure reforms to prevent settlement of the solid particles.

The invention is not limited as to the manner in which the dispersing agent and the structuring agent are added. The structuring agent may for instance be incorporated in the aqueous mineral pigment suspension at the same time as the dispersing agent or at some later stage. Alternatively the dispersing agent may be incorporated in the aqueous suspension after classification to remove coarse material and dewatering but before comminution, the structuring agent being added after the suspension has been subjected to comminution.

The invention is illustrated by the preparation of a slurry suitable for use in coating compositions, as follows.

In the production of a fine grade whiting from naturally occurring chalk, the chalk will be formed into a suspension containing 30 per cent to 50 per cent by weight of water and this will be freed from coarse hard particles of flint and the like by conventional wet classification followed by dewatering by settling or centrifuging, and filtration to provide a cake which will usually contain an amount of water in the range of 15 per cent to 35 per cent by weight. The small additions of dispersing agent and structuring agent may then together be incorporated in the filter cake by a simple mixing device and the resulting fluid suspension can then be pumped through a mill to effect the necessary particle size reduction. For a cake containing about 30 per cent water, and containing not more than 0.5 per cent by weight of particles above 45  $\mu\text{m}$  and 70 per cent by weight less than 2  $\mu\text{m}$ , there may for example be used 0.075 per cent of Tetron (sodium

polyphosphate) as dispersing agent and 0.08 per cent of Laponite (synthetic hectorite) as structuring agent. Alternatively the filter cake may be treated with the dispersing agent alone and the structuring agent be added separately after the milling operation which method may be preferable if there is any possibility that the shear rate might not be sufficient to completely neutralise the effect of the structuring agent in the milling operation. In either case the suspension obtained will be sufficiently stabilised to prevent settling out during transport and storage for up to at least 30 days and will be suitable for use in the manufacture of water-borne surface coating compositions designed for application to paper and the like or to interior or exterior wall surfaces, ceilings, etc. A similar suspension containing 0.4 per cent dispersing agent but no structuring agent would, by comparison, settle in about 20 days.

## 85 CLAIMS

1. A method of rendering readily pumpable but stable an aqueous suspension of mineral particles having a water content of not more than 40 per cent by weight of the suspension, by incorporating in the suspension a structuring agent as herein defined.

2. A method according to Claim 1 wherein the water content is not more than 35 per cent.

3. A method according to Claim 1 or 2, which comprises also incorporating in the suspension a dispersing agent capable of reducing the viscosity of aqueous slurries of particulate minerals.

4. A method according to Claim 1, 2 or 3, wherein the size distribution of the mineral particles is such that not more than 0.5 per cent by weight of particles is retained on a 45  $\mu\text{m}$  mesh sieve and at least 25 per cent by weight of the particles are of size less than 2  $\mu\text{m}$  equivalent spherical diameter.

5. A method according to Claim 4 wherein at least 60 per cent by weight of the particles are of size less than 2  $\mu\text{m}$  equivalent spherical diameter.

6. A method according to any preceding Claim wherein the mineral particles are of chalk whiting, kaolin or limestone.

7. A method according to any preceding Claim wherein the structuring agent is selected from synthetic hectorites, titanium esters, silica sols, polyvinyl alcohols of molecular weight 14000 to 125000, carboxymethyl cellulose, casein and quaternary ammonium salts.

8. A method according to any preceding Claim wherein the structuring agent is incorporated in the suspension in an amount of 0.01 to 1.5 per cent by weight based on the weight of the solid particles.

9. A method according to any of Claims 3 to 9, wherein the dispersing agent is selected from water soluble salts of polyacrylic and polymethacrylic acids, water soluble polyphosphates and polysilicates, lignosulphonates and alkyl aryl sulphonates.

10. A method according to any of Claims 3 to 9, wherein the dispersing agent is incorporated in

the suspension in an amount of 0.01 to 1.0 per cent by weight based on the weight of the solid particles.

- 5 11. A method according to Claim 1 substantially as described in the foregoing illustrative example.

12. An aqueous mineral pigment suspension produced by the method of any preceding Claim.

- 10 13. A paper coating composition containing mineral pigment in which all or part of the mineral pigment content is provided in the form of a suspension as claimed in Claim 12.